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**Title:  
ALPHA-ALUMINA.**

**Abstract:**

A powdery alpha -alumina comprising single-crystal alpha -alumina grains which are homogeneous and free from any crystal seed inside, form an at least eight-sided polyhedron, and have a D to H ratio of 0.5 to 3.0 (wherein D is the maximum grain diameter in parallel with the hexagonal lattice plane of the alpha -alumina having a hexagonal closest lattice structure and H is the grain diameter perpendicular to the hexagonal lattice plane thereof), a number-average grain diameter of 0.1 to 5  $\mu$ m and a narrow grain size distribution. The powdery alpha -alumina of the invention comprises single-crystal alpha -alumina grains which are nearly spherical, fine and structurally homogeneous and have a narrow grain size distribution, and is suitable as the raw materials of abrasive, sinter, plasma spraying material, filler, single crystal, catalyst support, phosphor, sealing material and ceramic filter, in particular, as the raw materials of precision abrasive, sinter and ceramic filter, thus being remarkably useful from the industrial viewpoint.



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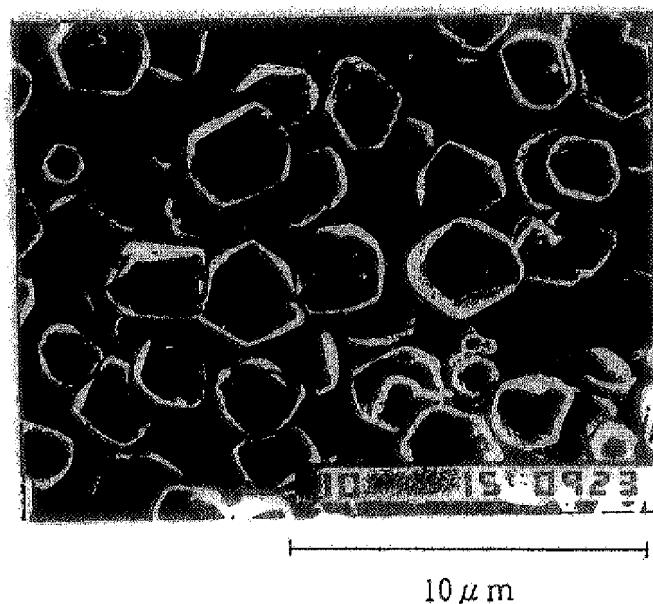
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㉕ ALPHA-ALUMINA.

㉖ A powdery  $\alpha$ -alumina comprising single-crystal  $\alpha$ -alumina grains which are homogeneous and free from any crystal seed inside, form an at least eight-sided polyhedron, and have a  $D_{10}/D_{90}$  ratio of 0.5 to 0.9 (wherein D is the maximum grain diameter in parallel with the hexagonal lattice plane of the  $\alpha$ -alumina having a hexagonal closest lattice structure and H is the grain diameter perpendicular to the hexagonal lattice plane thereof), a number-average grain diameter of 0.1 to 6  $\mu\text{m}$  and a narrow grain size distribution. The powdery  $\alpha$ -alumina of the invention comprises single-crystal  $\alpha$ -alumina grains which are nearly spherical, fine and structurally homogeneous and have a narrow grain size distribution, and is suitable as the raw materials of abrasive, sinter, plasma spraying material, filler, single crystal, catalyst support, phosphor, sealing material and ceramic filter, in particular, as the raw materials of precision abrasive, sinter and ceramic filter, thus being remarkably useful from the industrial viewpoint.

Fig. 1



TECHNICAL FIELD

This invention relates to  $\alpha$ -alumina.  $\alpha$ -Alumina powder has been widely used as an abrasive, a raw material for sintered products, a plasma spraying material, a filler and the like. The  $\alpha$ -alumina of the present invention comprises  $\alpha$ -alumina single crystal particles which are not agglomerated particles and have high purity, structural homogeneity, and a narrow particle size distribution, and is of high industrial use as an abrasive, a raw material for sintered products, a plasma spraying material, a filler, a starting material for single crystals, a raw material for a carrier for catalysts, a raw material for fluorescent substances, a raw material for encapsulations, a raw material for ceramic filters, etc. It is particularly useful as a precision abrasive, a raw material for sintered products, and a raw material for ceramic filters.

BACKGROUND ART

$\alpha$ -Alumina powder obtained by conventional processes comprises irregular-shaped polycrystals, contains many agglomerates, and has a broad particle size distribution. For some uses, the purity of these conventional species is insufficient. In order to eliminate these problems,  $\alpha$ -alumina powder produced by special processes as hereinafter described has been employed for specific uses. However, these special processes still fail to arbitrarily control the shape or particle diameter of  $\alpha$ -alumina powder. It has thus been difficult to obtain  $\alpha$ -alumina powder having a narrow particle size distribution.

Known special processes for producing  $\alpha$ -alumina powder include a process utilizing a hydrothermal reaction of aluminum hydroxide (hereinafter referred to as hydrothermal treatment process); a process comprising adding a flux to aluminum oxide, fusing, and precipitating (hereinafter referred to as flux process); and a process in which aluminum hydroxide is calcined in the presence of a mineralizer.

With respect to a hydrothermal treatment process, JP-B-57-22886 (the term "JP-B" as used herein means an "examined published Japanese patent application") discloses a process in which corundum is added as a seed crystal to control the particle size. The process consists of synthesis in a high temperature under a high pressure, making the resulting  $\alpha$ -alumina powder expensive.

According to the study by Matsui, et al. (Hydrothermal Hannou (Hydrothermal Reactions)), Vol. 2, pp. 71-78 "Growth of Alumina Single Crystal by Hydrothermal Methods"), an  $\alpha$ -alumina single crystal obtained by growth of an alumina single crystal containing chromium on a sapphire ( $\alpha$ -alumina) seed crystal by a hydrothermal growth process (hydrothermal treatment process) contains cracks. On examining the homogeneity of the crystal in an attempt to clarify the cause of the cracks, it was confirmed that a high strain exists in the boundary between the seed crystal and the grown crystal and that the density of etch pit in the grown crystal near the boundary, which seems to correspond to the dislocation density, is high. The report goes that the cracks are expected to relate to such a strain or a defect and that a hydrothermal growth process is apt to involve incorporation of a hydroxyl group or water into crystals, which appears to cause a strain or a defect.

A flux process has been proposed as a means for controlling the shape or particle size of  $\alpha$ -alumina powder for use as an abrasive, a filler, etc. For example, JP-A-3-131517 (the term "JP-A" as used herein means an "unexamined published Japanese patent application") discloses a process comprising calcining aluminum hydroxide in the presence of a flux with fluorine having a melting point of not more than 800°C to prepare  $\alpha$ -alumina powder having an average particle size of from 2 to 20  $\mu\text{m}$  and a hexagonal plate shape having a D/H ratio of from 5 to 40, wherein D represents a maximum particle diameter parallel to a hexagonal lattice plane of a hexagonal close-packed lattice of  $\alpha$ -alumina, and H represents a diameter perpendicular to the hexagonal lattice plane. However, this process cannot provide fine  $\alpha$ -alumina powder having a particle diameter of not more than 2  $\mu\text{m}$ , and all the particles obtained have a plate shape. Therefore, the resulting  $\alpha$ -alumina powder is not always suitable for use as an abrasive, a filler and a starting material for single crystals.

The Bayer process is a commonly employed and the least expensive process for producing  $\alpha$ -alumina powder. In the Bayer process, bauxite is once converted to aluminum hydroxide or transition alumina, which is then calcined in air to prepare  $\alpha$ -alumina powder.

The aluminum hydroxide or transition alumina which is obtained as an intermediate product on an industrial scale at low cost comprises large agglomerates having a particle diameter of greater than 10  $\mu\text{m}$ . Conventional  $\alpha$ -alumina powder obtained by calcination of such aluminum hydroxide or transition alumina in air comprises particles of irregular shape containing agglomerated coarse particles. The  $\alpha$ -alumina powder containing agglomerated coarse particles are ground into final products by means of a ball mill, a vibration mill, etc., but grinding is not always easy and incurs the cost.  $\alpha$ -Alumina powder having difficulty in grinding needs an extended period of time for grinding, during which fine powder may be formed or foreign

materials may be incorporated only to provide  $\alpha$ -alumina powder unsuitable as an abrasive.

Several proposals have been made to date as a solution to these problems. For example, JP-A-59-97528 discloses a process for producing  $\alpha$ -alumina powder with an improved shape comprising calcining aluminum hydroxide prepared by the Bayer process in the presence of boron-containing ammonium and a 5 boron mineralizer to obtain  $\alpha$ -alumina powder having an average particle diameter of from 1 to 10  $\mu\text{m}$  and a D/H ratio approximate to 1. However, this process involves problems in that the boron-containing or fluorine-containing material added as a mineralizer remains in the resulting  $\alpha$ -alumina and agglomerates are formed upon calcining.

In connection to calcination of sodium-containing aluminum hydroxide prepared by the Bayer process, it 10 has been proposed to conduct calcining in the presence of a fluoride, e.g., aluminum fluoride or cryolite, and a chlorine-containing material, e.g., chlorine or hydrogen chloride in British Patent 990,801; or in the presence of boric acid, and ammonium chloride, hydrochloric acid or aluminum chloride in West German Patent 1,767,511 for the purpose of effectively removing sodium while controlling the particle diameter.

However, in the former process, since a mineralizer such as aluminum fluoride is added in a solid form 15 or the calcination is conducted while supplying chlorine gas and fluorine gas without the addition of water, the resulting alumina particles have problems of an irregular shape and a broad particle size distribution. The latter process also involves a problem in that boric acid as a mineralizer remains in the resulting  $\alpha$ -alumina in the form of a boron-containing material. In addition, these processes aim chiefly at removal of 20 sodium, and the sodium salt, such as  $\text{NaCl}$  or  $\text{Na}_2\text{SO}_4$ , by-produced by the reaction between sodium and a sodium removing agent must be sublimed or decomposed by calcination at a high temperature of at least 1,200 °C.

With respect to the reaction between alumina and hydrogen chloride gas, there is a report in Zeit. fur Anorg. und Allg. Chem., Vol 21, p. 209 (1932) of an equilibrium constant of the reaction system comprising sintered  $\alpha$ -alumina having a particle diameter of from 2 to 3 mm, hydrogen chloride, and produced 25 aluminum chloride. According to the report, while  $\alpha$ -alumina is found produced in a place different from the place where the starting material has been charged, only hexagonal plate-shaped particles are obtained.

JP-B-43-8929 discloses a process comprising calcining alumina hydrate in the presence of ammonium chloride to produce alumina having a low impurity content and an average particle diameter of not more than 10  $\mu\text{m}$ . The resulting alumina powder has a broad particle size distribution.

30 Therefore, none of the conventional techniques succeeded in providing  $\alpha$ -alumina single crystal particles which comprises fine and non-agglomerated  $\alpha$ -alumina single crystals particles and is particularly suitable as a precision abrasive, a raw material for sintered products or a raw material for ceramic filters.

An object of the present invention is to solve the above problems and to obtain  $\alpha$ -alumina in a powder form which comprises fine, homogeneous, and non-agglomerated  $\alpha$ -alumina single crystal particles. More 35 specifically, it is to provide  $\alpha$ -alumina in a powder form comprising  $\alpha$ -alumina single crystal particles which have an octa- or higher polyhedral shape, a D/H ratio of from 0.5 to 3.0, a narrow particle size distribution, a high alumina purity, and uniform composition within the particle, with the individual particles being freed of structural strain.

#### 40 DISCLOSURE OF THE INVENTION

The present invention relates to the following inventions.

(1)  $\alpha$ -Alumina characterized in that it comprises  $\alpha$ -alumina single crystal particles which are homogeneous containing no crystal seed inside the particles, have an octa- or higher polyhedral shape, and have a D/H ratio of from 0.5 to 3.0, wherein D represents a maximum particle diameter parallel to a hexagonal lattice plane of a hexagonal close-packed lattice of the particles, and H represents a diameter perpendicular to the hexagonal lattice plane; and the  $\alpha$ -alumina has a number average particle diameter of from 100 to 500  $\mu\text{m}$ .

(2)  $\alpha$ -Alumina as described in (1) above, wherein the  $\alpha$ -alumina has such a particle size distribution that the D90/D10 ratio is not more than 10, wherein D10 and D90 represents a cumulative 10% diameter and a cumulative 90% diameter, respectively, of a cumulative distribution depicted from the small diameter side.

(3)  $\alpha$ -Alumina as described in (1) or (2) above, wherein the  $\alpha$ -alumina has a number average particle diameter of from 100 to 500  $\mu\text{m}$ .

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 is a scanning electron microscope (SEM) photograph (magnification: 4,900) showing the particulate shape of the  $\alpha$ -alumina obtained in Example 8.

5 Fig. 2 is a particle size distribution of the  $\alpha$ -alumina obtained in Example 8.

Fig. 3 is an SEM photograph (magnification: 930) showing the particulate shape of the  $\alpha$ -alumina obtained in Example 4.

10 Fig. 4 is an SEM photograph (magnification: 1,900) showing the particulate shape of the  $\alpha$ -alumina obtained in Example 2.

Fig. 5 is an SEM photograph (magnification: 1,900) showing the particulate shape of the  $\alpha$ -alumina obtained in Comparative Example 2.

15 Fig. 6 is an SEM photograph (magnification: 930) showing the particulate shape of the  $\alpha$ -alumina obtained in Comparative Example 3.

Fig. 7 shows the crystal habit of an  $\alpha$ -alumina single crystal.

BEST MODE FOR PRACTICING THE INVENTION

The present invention is described in detail below.

20 The  $\alpha$ -alumina according to the present invention can be prepared from transition alumina or a raw material capable of being converted to transition alumina on heating. Transition alumina means all alumina crystal phases included under polymorphic alumina represented by  $\text{Al}_2\text{O}_3$  except  $\alpha$ -alumina, specifically including  $\gamma$ -alumina,  $\delta$ -alumina,  $\theta$ -alumina, etc.

25 The raw material converted to transition alumina on heating includes those which are once converted to transition alumina and then to  $\alpha$ -alumina by calcination, such as aluminum hydroxide, aluminum sulfate, alum (e.g., potassium aluminum sulfate, ammonium aluminum sulfate), ammonium aluminum carbonate hydroxide, and alumina gel, e.g., alumina gel obtained by electro-discharge in water.

30 Synthesis methods of the transition alumina and the raw material converted to transition alumina on heating are not particularly limited. Aluminum hydroxide, for example, can be obtained by the Bayer process, hydrolysis of an organoaluminum compound, or a process using, as a starting material, an aluminum compound recovered from an etching waste used for condensers, etc.

35 Transition alumina can be obtained by a heat treatment of aluminum hydroxide, decomposition of aluminum sulfate, decomposition of alum, vapor phase decomposition of aluminum chloride, or decomposition of ammonium aluminum carbonate.

40 The transition alumina or the raw material converted to transition alumina on heating is calcined in an atmosphere containing at least 1% by volume, preferably at least 5% by volume, and more preferably at least 10% by volume, of ~~hydrogen chloride~~ gas. Gases for dilution of hydrogen chloride gas include inert gases, e.g., nitrogen and argon, hydrogen and air. The pressure of the hydrogen chloride-containing atmosphere is not particularly limited and is selected arbitrarily from an industrially practical range.  $\alpha$ -Alumina in a powder form having the excellent properties as desired can thus be obtained by calcination in a relatively low temperature as hereinafter described.

45 Hydrogen chloride gas may be replaced with a mixed gas of chlorine and steam. In this case, transition alumina or the raw material converted to transition alumina on heating is calcined while at least 1% by volume, preferably at least 5% by volume, and more preferably at least 10% by volume, of chlorine gas and at least 0.1% by volume, preferably at least 1% by volume, and more preferably at least 5% by volume, of steam are introduced to an atmosphere. Gases for diluting the mixed gas of chlorine and steam include inert gases, e.g. nitrogen and argon, hydrogen and air. The pressure of the chlorine- and steam-containing atmosphere is not particularly limited and is selected arbitrarily from an industrially practical range.  $\alpha$ -Alumina in a powder form having the excellent properties as desired can thus be obtained by calcining in a relatively low temperature as hereinafter described.

50 The calcining temperature is generally 600 °C or higher, preferably from 600 to 1,400 °C, more preferably from 700 to 1,300 °C, and most preferably from 800 to 1,200 °C. By calcining at a temperature controlled within this range,  $\alpha$ -alumina in a powder form comprising  $\alpha$ -alumina single crystal particles which are hardly agglomerated and, even immediately after calcination, show a narrow particle size distribution can be obtained at an industrially advantageous rate of formation.

55 A proper calcining time depends on the concentration of the gas in the calcining atmosphere and the calcining temperature but is preferably 1 minute or more, and more preferably 10 minutes or more. The calcining time is sufficient if the alumina raw material undergoes crystal growth to form  $\alpha$ -alumina. Desired  $\alpha$ -alumina can be obtained in a shorter calcining time than required in the conventional processes.

The source and the mode of supplying the gas of the calcining atmosphere are not particularly restricted as long as the chlorine-containing gas is introduced into the reaction system containing the starting material. For example, the component gases may be supplied from gas cylinders. Where a chlorine compound, e.g., hydrochloric acid solution, ammonium chloride, or a chlorine-containing high polymer is used as a source for the chlorine gas, it is used at its vapor pressure or as decomposed so as to give a prescribed gas composition. In some cases of using decomposition gas of ammonium chloride, etc., a solid substance deposits in a calcining furnace to cause operational troubles. Further, as the hydrogen chloride gas concentration increases, the calcination can be effected at a lower temperature in a shorter time, and the purity of the resulting  $\alpha$ -alumina can be increased. Accordingly, it is preferable to supply hydrogen chloride or chlorine directly from a gas cylinder into a calcining furnace. The gas supply may be either in a continuous manner or in a batch system.

The calcining apparatus is not particularly limited, and a conventional calcining furnace can be employed. The calcining furnace is preferably made of a material resistant to corrosion by hydrogen chloride gas, chlorine gas, etc. The furnace is preferably equipped with a mechanism for controlling the atmosphere. Because an acid gas, e.g., hydrogen chloride or chlorine gas, is used, the furnace is preferably air-tight. For industrial production, calcination is preferably carried out in a continuous manner by means of, for example, a tunnel kiln, a rotary kiln, a pusher kiln, etc.

Since the reaction proceeds in an acidic atmosphere, a crucible, a boat or a like tool used in the process is preferably made of alumina, quartz, acid resistant brick, or graphite.

The  $\alpha$ -alumina according to the present invention which is not agglomerated particles can thus be obtained. Depending on the starting material or the conditions of calcination, the resulting  $\alpha$ -alumina may be agglomerated particles or may contain agglomerated particles. Even in such cases, the degree of agglomeration is very slight, and simple grinding will provide  $\alpha$ -alumina having the above-mentioned excellent characteristics according to the present invention.

In order to obtain  $\alpha$ -alumina in a powder form of the present invention which has a number average particle diameter of from 0.1 to 5  $\mu\text{m}$ , it is preferred to use a raw material having a high alumina purity of from 99.5 to 99.9% by weight. Specific examples thereof include aluminum hydroxide powder obtained by the Bayer method, and transition alumina and alum obtained from the aluminum hydroxide powder.

The  $\alpha$ -alumina single crystal particles constituting the  $\alpha$ -alumina of the present invention have excellent properties, i.e., a number average particle diameter of from 0.1 to 5  $\mu\text{m}$ , a D/H ratio of from 0.5 to 3.0, and a D90/D10 ratio of not more than 10, preferably not more than 9, and more preferably not more than 7, wherein D10 and D90 represent a cumulative 10% diameter and a cumulative 90% diameter, respectively of cumulative distribution depicted from the small diameter side.

## 35 EXAMPLE

The present invention will now be illustrated in greater detail with reference to Examples, but it should be understood that the present invention is not construed as being limited thereto.

Various measurements in Examples and Comparative Examples were made as follows.

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### 1. Particle Diameter and Particle Diameter Distribution of $\alpha$ -Alumina:

- (1) A D90/D10 ratio was measured by the laser scattering method with "Master Sizer" manufactured by Malvern Instruments, Ltd.
- (2) A micrograph of  $\alpha$ -alumina was taken with an SEM ("T-300" manufactured by Japan Electron Optics Laboratory Co., Ltd., hereinafter the same), and selected 80 to 100 particles of the SEM photograph were subjected to image analysis to obtain an average and a distribution of their circle-equivalent diameters. The term "circle-equivalent diameter" as used herein means a diameter of a true circle having the same area as a particle.

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### 2. Crystal Shape (D/H) of $\alpha$ -Alumina:

The shape of  $\alpha$ -alumina particles was represented in terms of a D/H ratio, wherein D and H are as defined above. A D/H ratio of  $\alpha$ -alumina was obtained as an average of 5 to 10 particles by image analysis of the above-mentioned SEM photograph.

3. Number of Crystal Faces and Crystal Habit:

(1) The number of crystal faces of  $\alpha$ -alumina was obtained by observation of the above-mentioned SEM photograph.

5 (2) The crystal habit of  $\alpha$ -alumina particles was observed for evaluating the shape. The crystal habit of the  $\alpha$ -alumina particles obtained in the present Invention (indicated by A to I) is shown in Fig. 7.  $\alpha$ -Alumina has a hexagonal system, and the term "crystal habit" used for  $\alpha$ -alumina means the form of its crystal characterized by the appearance of the crystal faces composed of a plane {1120}, c plane {0001}, n plane {2243}, and r plane {1012}. In Fig. 7 are shown crystal planes a, c, n, and r.

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4. Alumina Purity:

The amount of ions of incorporated impurities was measured by emission spectrochemical analysis to obtain the content of the impurities on oxide conversion. The chlorine content was measured by potentiometry. An alumina purity was obtained by subtracting the total impurity content (wt%) thus calculated from 100% by weight.

5. Na<sub>2</sub>O Content:

20 The amount of a sodium ion incorporated was measured by emission spectrochemical analysis to obtain the content of Na<sub>2</sub>O.

Raw materials used in Examples were as follows.

## 1. Transition Alumina A:

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Transition alumina obtained by calcination of aluminum hydroxide prepared by hydrolysis of aluminum isopropoxide ("AKP-G15" produced by Sumitomo Chemical Co., Ltd.; secondary particle diameter: about 4  $\mu\text{m}$ )

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## 2. Transition Alumina C:

Transition alumina obtained by calcination of aluminum hydroxide C (described hereinafter) in air at 800 °C (secondary particle diameter: about 30  $\mu\text{m}$ )

## 3. Aluminum Hydroxide B:

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Aluminum hydroxide powder prepared by the Bayer process ("C 301" produced by Sumitomo Chemical Co., Ltd.; secondary particle diameter: about 2  $\mu\text{m}$ )

## 4. Aluminum Hydroxide C:

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Aluminum hydroxide powder prepared by the Bayer process ("C 12" produced by Sumitomo Chemical Co., Ltd.; secondary particle diameter: about 47  $\mu\text{m}$ )

5. Alum (Al NH<sub>4</sub>(SO<sub>4</sub>)<sub>2</sub> 12H<sub>2</sub>O):

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Transition alumina precursor which gives transition alumina on heating. The reagent of Wako Pure Chemical Industries, Ltd. was used.

Hydrogen chloride in a cylinder produced by Tsurumi Soda K.K. (purity: 99.9%) was used as a hydrogen chloride gas source, and chlorine in a cylinder produced by Fujimoto Sangyo K.K. (purity: 99.4%) was used as a chlorine gas source. The volume percent of steam was controlled by adjusting the saturated vapor pressure of water dependent on temperature and introduced into a furnace with nitrogen gas.

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An alumina boat was filled with 0.4 g of a raw material, such as transition alumina or aluminum hydroxide, to a depth of 5 mm. Calcination of the raw material was conducted in a tube furnace ("DSPSH-28" manufactured by Motoyama K.K.) using a quartz tube (diameter: 27 mm; length: 1,000 mm). The temperature was increased at a rate of 500 °C/hour while introducing nitrogen, and hydrogen chloride gas or a mixed gas of chlorine and steam was introduced into the furnace when a prescribed temperature was reached.

The gas concentration was controlled by adjustment of the gas flow rate by means of a flowmeter. The linear flow rate of the gas was adjusted between 20 and 40 mm/min. This system was hereinafter referred

to as a gas flow system. The total pressure of the atmospheric gases was atmospheric pressure.

On reaching a prescribed temperature, the furnace was maintained at that temperature (hereinafter referred to as a calcining temperature) for a prescribed time (hereinafter referred to as a keeping time). After a lapse of a prescribed keeping time, the furnace was allowed to cool to obtain  $\alpha$ -alumina in a powder form.

The steam partial pressure was controlled by adjusting the saturated vapor pressure, and steam was fed to the furnace with nitrogen gas.

#### EXAMPLES 1 TO 6

Aluminum hydroxide or transition alumina ( $\gamma$ -alumina) was calcined in a hydrogen chloride gas atmosphere at a calcining temperature of 1,100 °C or 900 °C.

The SEM photographs of the  $\alpha$ -alumina obtained in Examples 2 and 4 are shown in Figs. 4 and 3, respectively. The calcining conditions and the results are shown in Tables 1 and 2, respectively.

#### EXAMPLE 7

Calcination of aluminum hydroxide was carried out under the same conditions as in Example 3, except for using an atmosphere containing chlorine gas and steam and changing the gas flow rate as shown in Table 1. The results obtained are shown in Table 2.

#### EXAMPLE 8

Calcination was carried out under the same conditions as in Example 3, except for using alum as a raw material. The results obtained are shown in Table 2. The SEM photograph and the particle diameter distribution of the resulting  $\alpha$ -alumina in a powder form are shown in Figs. 1 and 2, respectively.

#### COMPARATIVE EXAMPLES 1 AND 2

Aluminum hydroxide C was calcined in air according to the conventional process. The calcining conditions and the results obtained are shown in Tables 1 and 2. The SEM photograph of the  $\alpha$ -alumina in a powder form obtained in Comparative Example 2 is shown in Fig. 5.

#### COMPARATIVE EXAMPLE 3

Transition alumina A was calcined in an atmosphere having a hydrogen chloride gas concentration as low as 0.5% by volume. The calcining conditions and the results are shown in Tables 1 and 2. The SEM photographs of the resulting alumina in a powder form is shown in Fig. 6.

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50      51      52      53      54      55      56      57      58      59      60      61

TABLE 1

	Alumina Raw Material Kind	Size ( $\mu\text{m}$ )	Composition of Atmosphere (vol %)				Gas Flow Rate (mm/min)	Temp. for Gas Introduction ( $^{\circ}\text{C}$ )	Calcin- ing Temp. ( $^{\circ}\text{C}$ )	Keeping Time (min)
			HCl	Cl <sub>2</sub>	H <sub>2</sub> O	H <sub>2</sub>				
Example 1	transition alumina C	30	100	-	-	-	35	700	1,100	30
Example 2	aluminum hydroxide B	2	33	-	-	17	50	20	800	1,100
Example 3	aluminum hydroxide C	47	30	-	-	70	-	20	20	1,100
Example 4	aluminum hydroxide C	47	100	-	-	-	-	35	20	1,100
Example 5	aluminum hydroxide C	47	30	-	-	70	-	35	500	1,100
Example 6	aluminum hydroxide C	47	100	-	-	-	-	35	20	900
Example 7	aluminum hydroxide C	47	-	35	5	60	-	49	20	1,100
Example 8	alum	-	30	-	-	70	-	20	20	1,100
Comparative Example 1	aluminum hydroxide C	47	calcination in air				-	-	1,300	180
Comparative Example 2	aluminum hydroxide C	47	calcination in air				-	-	1,100	180
Comparative Example 3	transition alumina A	4	0.5	-	-	99.5	-	0	20	1,100

TABLE 2

	Number Average Particle Diameter (μm)	Crystal Shape Crystal Habit D/H	Number of Crystal Faces	Size Distribution D90/D10	Alumina Purity (wt%)	Na <sub>2</sub> O Content (ppm)	Remark
Example 1	4	1 - 2	C, A	14 or more	-	-	-
Example 2	2	1 - 2	-	14 or more	9	>99.95	-
Example 3	4	2	-	20 or more	9	-	-
Example 4	4	1 - 2	F, H	8 or more	7	99.95	-
Example 5	4	1 - 2	C	14 or more	-	-	-
Example 6	4	1 - 2	C, I	8 or more	-	-	-
Example 7	5	1 - 2	C, A	8 or more	-	>99.90	-
Example 8	2	1	F, H	20 or more	9	-	-
Comparative Example 1	0.3	no formation of single crystal	-	-	-	-	α-crystal
Comparative Example 2	-	no formation of single crystal	-	-	-	-	γ-crystal
Comparative Example 3	-	no formation of single crystal	-	-	-	-	θ-crystal

## INDUSTRIAL UTILITY

The α-alumina according to the present invention can be obtained from raw materials of various kinds, purities, shapes, sizes, and compositions. The α-alumina of the present invention comprises octahedral or higher polyhedral α-alumina single crystal particles containing no crystal seed therein, having high purity,

homogeneity, and a narrow particle size distribution, and containing no agglomerates.

More specifically, the single crystal particles constituting the  $\alpha$ -alumina of the present invention have a number average particle diameter of from 0.1 to 5  $\mu\text{m}$ , and a D/H ratio of from 0.5 to 3.0, a D90/D10 ratio of not more than 10, wherein D10 and D90 represent a cumulative 10% diameter and a cumulative 90% diameter, respectively of cumulative distribution depicted from the small diameter side.

The  $\alpha$ -alumina powder comprising  $\alpha$ -alumina single crystal particles of high purity, uniform structure and narrow particle size distribution is suitable as an abrasive, a raw material for sintered products, a plasma flame spraying material, a filler, a raw material for single crystals, a raw material for a carrier of catalysts, a raw material for fluorescent substances, a raw material for encapsulations, a raw material for ceramic filters, etc. and is particularly useful as a precise abrasive, a raw material for sintered products, and a raw material for ceramic filters.

### Claims

1.  $\alpha$ -Alumina characterized in that it comprises  $\alpha$ -alumina single crystal particles which are homogeneous containing no crystal seed inside said particles, have an octa- or higher polyhedral shape, and have a D/H ratio of from 0.5 to 3.0, wherein D represents a maximum particle diameter parallel to a hexagonal lattice plane of a hexagonal close-packed lattice of said particles, and H represents a diameter perpendicular to said hexagonal lattice plane; and said  $\alpha$ -alumina has a number average particle diameter of from 0.1 to 5  $\mu\text{m}$ .
2.  $\alpha$ -Alumina as in Claim 1, wherein said  $\alpha$ -alumina has such a particle size distribution that the D90/D10 ratio is not more than 10, wherein D10 and D90 represents a cumulative 10% diameter and a cumulative 90% diameter, respectively, of a cumulative distribution depicted from the small diameter side.
3.  $\alpha$ -Alumina powder as in Claim 1 or 2, wherein said  $\alpha$ -alumina has a number average particle diameter of from 0.5 to 3  $\mu\text{m}$ .

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Fig. 1

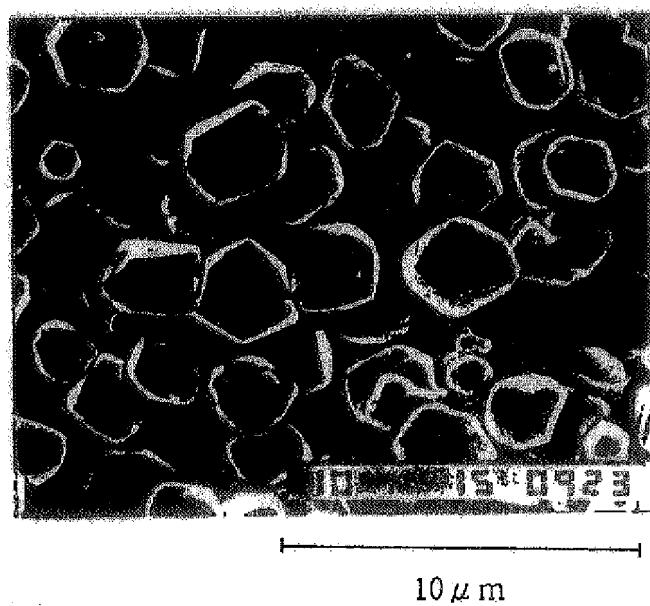


Fig. 2

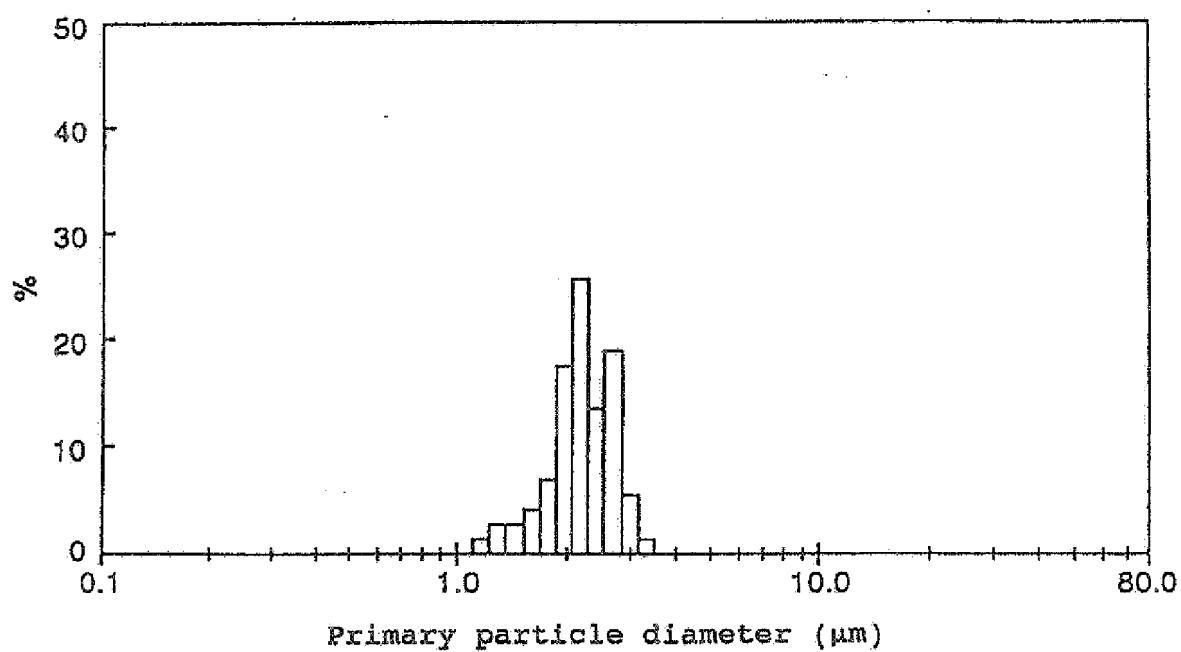


Fig. 3

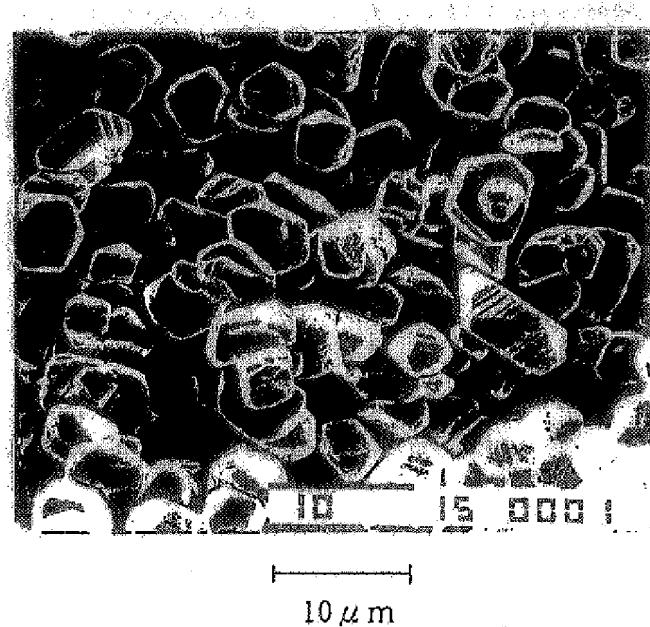


Fig. 4

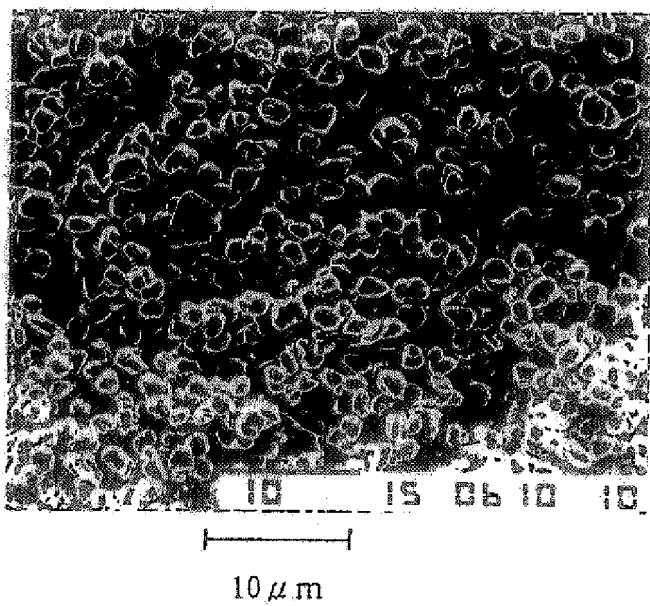


Fig. 5

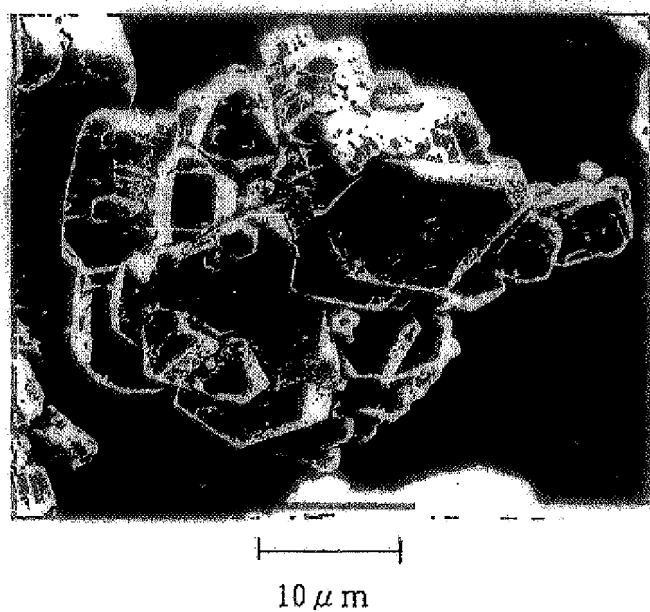


Fig. 6

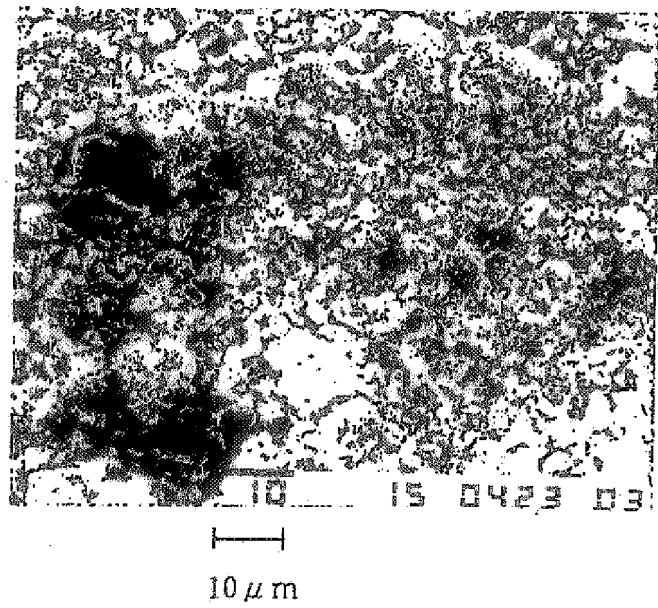
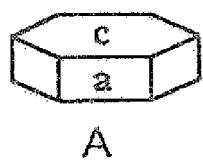
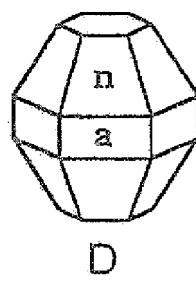


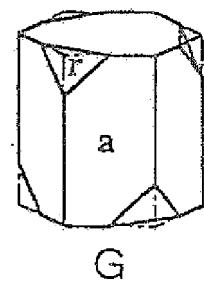
Fig. 7



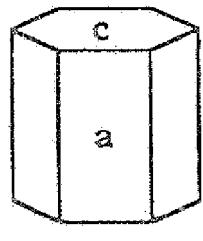
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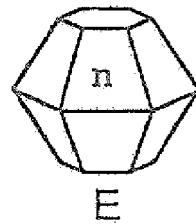
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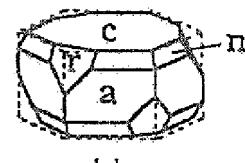
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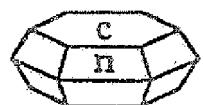
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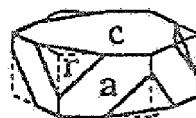
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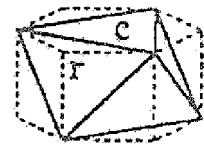
H



C



F



I

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP93/00739

## A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl<sup>5</sup> C30B29/20, C01F7/44

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

Int. Cl<sup>5</sup> C30B29/20, C01F7/44, C01F7/30

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho	1926 - 1993
Kokai Jitsuyo Shinan Koho	1971 - 1993

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP, A, 59-97528 (Swiss Aluminum Ltd.), June 5, 1984 (05. 06. 84), & EP, A, 105025	1-3
A	JP, A, 52-15498 (Showa Denko K.K.), February 5, 1977 (05. 02. 77), & US, A, 4193768	1-3
A	JP, A, 3-131517 (Atkem), June 5, 1991 (05. 06. 91), & FR, A, 2652075	1-3

 Further documents are listed in the continuation of Box C. See patent family annex.

\* Special categories of cited documents:

- "A" document defining the general state of the art which is not considered to be of particular relevance
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- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
- "Z" document member of the same patent family

Date of the search completion of the international search

August 10, 1993 (10. 08. 93)

Date of mailing of the international search report

August 31, 1993 (31. 08. 93)

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